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# Characterization of the Water Soluble Component of Inedible Residue from Candidate CELSS Crops

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December 1992

(NASA-TM-107557) CHARACTERIZATION  
OF THE WATER SOLUBLE COMPONENT OF  
INEDIBLE RESIDUE FROM CANDIDATE  
CELSS CROPS (Bionetics Corp.)  
21 p

N93-18111

Unclass

G3/54 0145779

National Aeronautics and  
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# **Characterization of the Water Soluble Component of Inedible Residue from Candidate CELSS Crops**

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**December 1992**



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## Abstract

Recycling of inorganic nutrients required for plant growth will be a necessary component of a fully closed, bioregenerative life support system. This research characterized the recovery of plant nutrients from the inedible fraction of three crop types (wheat, potato, and soybean) by soaking, or leaching, in water. A considerable portion of the dry weight of the inedible biomass was readily soluble (29% for soybean, 43% for wheat, and 52% for potato). Greater weight loss from potato was a result of higher tissue concentrations of potassium, nitrate, and phosphate. Approximately 25% of the organic content of the biomass was water soluble, while the majority of most inorganic nutrients, except for calcium and iron, was recovered in the leachate. Direct use of the leachates in hydroponic media could provide between 40-90% of plant nutrient demands for wheat, and 20-50% of demand for soybean and potato. Further evaluation of leaching as a component of resource recovery scheme in a bioregenerative system requires study of 1) utilization of plant leachates in hydroponic plant culture, and 2) conversion of organic material (both soluble and insoluble) into edible, or other useful, products.

## Introduction

Typical agroecosystems are based on nutrient flow into and out of the system (i.e. - application of fertilizer, removal of biomass). In order to reduce resupply requirements, agriculturally-based bioregenerative systems proposed for life support on long term space missions will require the recycling of nutrients contained in "waste" material (inedible plant parts, food processing wastes, human waste). These materials could be combusted or hydrolyzed to recycle organic carbon as CO<sub>2</sub> while providing an ash or other type of inorganic residue suitable for use in plant growth systems (Dreschel et al 1991, Takahashi et al. 1987, Modell 1986, Jacquez 1990). This approach would eliminate any potential production of edible products from the waste organic material through biological processing (e.g. - waste-based aquaculture systems, single cell protein production). Alternative, bioregenerative approaches would involve two separate processes: 1) biological conversion of waste organic material into edible products, and 2) production of an inorganic stream suitable for direct use in plant growth systems.

Previous research associated with the Controlled Ecological Life Support System (CELSS) Breadboard project at Kennedy Space Center (KSC) has indicated that the water soluble extract of inedible fraction of wheat may be suitable for use as a plant nutrient source in hydroponic systems. This water soluble fraction, or leachate, contains a significant percentage of the inorganic content of the biomass (Garland and Mackowiak 1989). Wheat growth and yield in leachate-based systems was similar to that in 1/2-strength Hoagland's solution in bench-top, recirculating hydroponic systems (Garland 1992). Controlled environment plant studies are currently being conducted to more fully examine the use of leachate-based nutrient solutions.

The leachate is a "mixed" fraction in that it contains both inorganic and organic molecules. Biological pretreatment of leachate prior to its use in hydroponic systems has been recommended to both reduce phytotoxic effects and to convert soluble

organics to edible products in regenerative systems (Garland and Mackowiak 1988, Garland 1992).

The present research characterizes the water soluble fraction of inedible residue from several candidate CELSS crops. Present knowledge of leachates from crops grown in controlled environments is largely limited to wheat. Several parameters important for the evaluation of leaching as a means of resource recovery were determined: 1) the percent recovery of nutrients from inedible biomass via leaching, 2) the relative concentrations of different nutrients in the leachate, particularly in comparison to the content of commonly used hydroponic solution, 3) the percentage of the plant nutrient requirements which could be supplied by leachate, and 4) the ratio of water soluble versus insoluble organic material.

## Materials and Methods

### Crop Material

The inedible fractions of mature wheat (*Triticum aestivum* L. cv. Yecora roja), white potato (*Solanum tuberosum* L. cv. Norland), and soybean (*Glycine max* L. Merr. cv. McCall) grown in the Biomass Production Chamber (BPC) at KSC were used in this study. Wheat biomass included chaff, leaves, stems, and roots. Potato biomass included leaves, stems, and non-tuberos roots. Soybean biomass included leaves, stems, roots, and seed pods. Crop biomass was oven-dried at 70 degrees C and ground through a 40 mesh screen prior to use. Three replicate leachings were performed using each type of crop material. Replicate wheat samples represented three different batches of a large pooled sample, while soybean and potato samples represented separate batches of biomass from different levels of the BPC.

### Leaching Conditions

Leaching was performed in a cylindrical (26.5 cm diameter, 30 cm height), steel vessel containing a removable wire mesh screen located above a bottom drain valve. A loading rate of 50 g biomass/ L deionized water was employed. Typically, leaching was done in 250 g batches with aeration (20 L/min) for two hours. A fraction of the liquid (approximately 1 L) was drained off, and added back to the top of the reactor (without disrupting the layer of residue retained on the screen) prior to collection of the entire liquid fraction, or leachate. This recycle step reduced the particulate load in the leachate by allowing the ground residue to form a filter "cake".

Preliminary experiments examined the effects of repeatedly rinsing the filter cake with additional batches of deionized water. The rinses were added to the top of the leaching vessels through a circular, perforated stainless steel tube. The spraying action of this water against the sides of the vessel allowed for rinsing of particulate matter adhered to the walls, while preventing disruption of the filter cake. The amount of nutrients removed from the filter cake was minimal after the fourth or fifth rinse (electrical conductivity less than 5% of the original rinse). Therefore, the leachate

described below was produced by mixing 50 g of crop material per L of deionized water, and rinsing with a equal volume of deionized water in five separate washings.

Leachate, as used here, cannot be strictly defined as the water soluble fraction of the inedible residue because a visible amount of fine particulate matter passes through the filter cake and screen. However, unfiltered leachate did not contain significantly greater concentrations of any element compared to filtered samples, indicating that this fine particulate fraction is not a significant source of nutrients. Results reported below are for unfiltered samples.

Dry weight of leached biomass was determined after drying for 24 hr at 70 degrees C. Ash content of unleached biomass was determined by combusting at 550 degrees C for two hours in a muffle furnace.

### Chemical Analysis

Samples of both leached and unleached biomass were analyzed for elemental content using an ion-coupled plasma (ICP) reactor by the Biomedical and Environmental Sciences Laboratory at the University of California at Los Angeles (UCLA). A common elemental scan was performed, and results of the major plant macro- and micronutrients are reported below with the exception of nitrogen for which no analysis was conducted. Aqueous leachate samples were also analyzed using an ICP at the KSC chemistry support lab. Dissolved nitrate and phosphate were assayed using a Technicon Autoanalyzer. Total organic carbon in leachate samples was measured using ultraviolet-assisted persulfate oxidation.

### Data Analysis

Percent nutrient recovery was calculated based on tissue analysis of unleached and leached biomass. The amount of material leached per unit weight of biomass was estimated from direct analysis of leachate samples.

### Results and Discussions

The weight loss for wheat (42%) found in this study (Table 1) is higher than the 35% reported by Garland and Mackowiak (1990). The percent recovery of several of the plant macronutrients is also higher (see below), indicating that one or several of the changes in leaching conditions in this study (grinding of wheat straw, mixing during leaching via aeration, rinsing with fresh deionized water) increased the solubilization of material.

### Nutrient Recovery from Leaching

Leaching as a nutrient removal process is effective; recovery was greater than 80% for several nutrients (K, Mg, Zn, and Cu) and greater than 60% for two others

Table 1. General characterization of the water soluble fraction of inedible residue from CELSS crops

	Crop Type		
	Wheat	Potato	Soybean
% Leachable (by weight)	42.6(1.0) <sup>1</sup>	51.8(0.5)	29.0(4.1)
% Ash	15.1(0.6)	25.8(0.4)	15.5(1.5)
Conductivity of Leachate (umhos/cm)	6290(113)	8800(196)	4180(196)
Total Organic Carbon Content of Leachate (mg/L)	1984(97)	2073(247)	1730(20)

1 - Values represent means and standard deviations of three replicates

(P and Mn) (Figure 1). Only two nutrients (Ca and Fe) were recovered at levels less than 50%. The percent recovery of N was not determined since nitrogen analysis was not performed on biomass samples. Based on the levels of nitrate in leachates, up to 10% of the inedible biomass is comprised of readily soluble nitrate. These very high estimates of nitrate in the leachate suggest that percent recovery is very high.

The efficiency of recovery was consistently different between crops for many of the nutrients, particularly P, Ca, and Fe. The percent recovery of calcium, for example, was approximately three times greater from wheat tissue (72%) than from potato tissue (23%). We have not investigated the underlying causes for these intercrop differences.

The overall quantity of water soluble components differed among the three crops examined, with a general ranking of potato > wheat > soybean (Table 1). This difference is present for both total weight loss, inorganic ions (i.e. - conductivity), and total organic carbon. The amount of all the macronutrients (except calcium) leached per gram of biomass is greater for potato than either soybean or wheat (Figure 2). The difference appears to be due to a higher tissue concentration of these elements rather than greater efficiency of removal since the percent recovery of nutrients from potato biomass is greater only for phosphorus (Figure 1).

Based on the weights of individual elements (i.e. - N, P, K, Ca, and Mg), 7, 10, and 15% of the dry weight of inedible residue from soybean, wheat, and potato, respectively, is comprised of soluble inorganic macronutrients. Potassium comprises between 68-75% of this inorganic weight. If the actual weights of the nutrients in their complete form are compared (i.e. - NO<sub>3</sub>, PO<sub>4</sub>, K, Ca, Mg), inorganic content accounts for 10, 17, and 25% of the dry weight of the inedible residue from soybean, wheat, and potato, respectively, and potassium accounts for 40-52% of the inorganic weight. Therefore, while the majority of the "waste" plant material is composed of organic elements (C and O), recycling approaches which recover inorganic elements and organic elements could reduce resupply requirements by 10-25% compared to those that recover organic elements alone.

The nutrient content of these hydroponically-grown crops is greater, on average, than that reported for field grown plants (Walsh and Beaton 1973). From a system perspective, a larger reservoir of nutrients within plant tissue could be considered detrimental because a greater absolute amount of nutrients need to be recycled through the biomass production system. The cost of recovering water soluble nutrients in the manner described here, however, is independent of the nutrient content of the biomass. Manipulation of nutrient solution composition so that elements are supplied to plants on a demand basis potentially could reduce the accumulation of nutrients in plant tissue, but would require a concomitant increase in the costs associated with monitoring and controlling nutrient concentration. In addition, plant growth would be more likely to be nutrient limited under such conditions. Providing "excess" nutrients would decrease the need for nutrient level control and buffer the plants from nutrient limitation. Therefore, a large reservoir of nutrients within plant tissue may be beneficial from the standpoint of overall system stability and efficiency as long as 1) nutrients can be readily recovered, and 2) tissue concentrations are kept within acceptable (i.e. - non-toxic) limits.

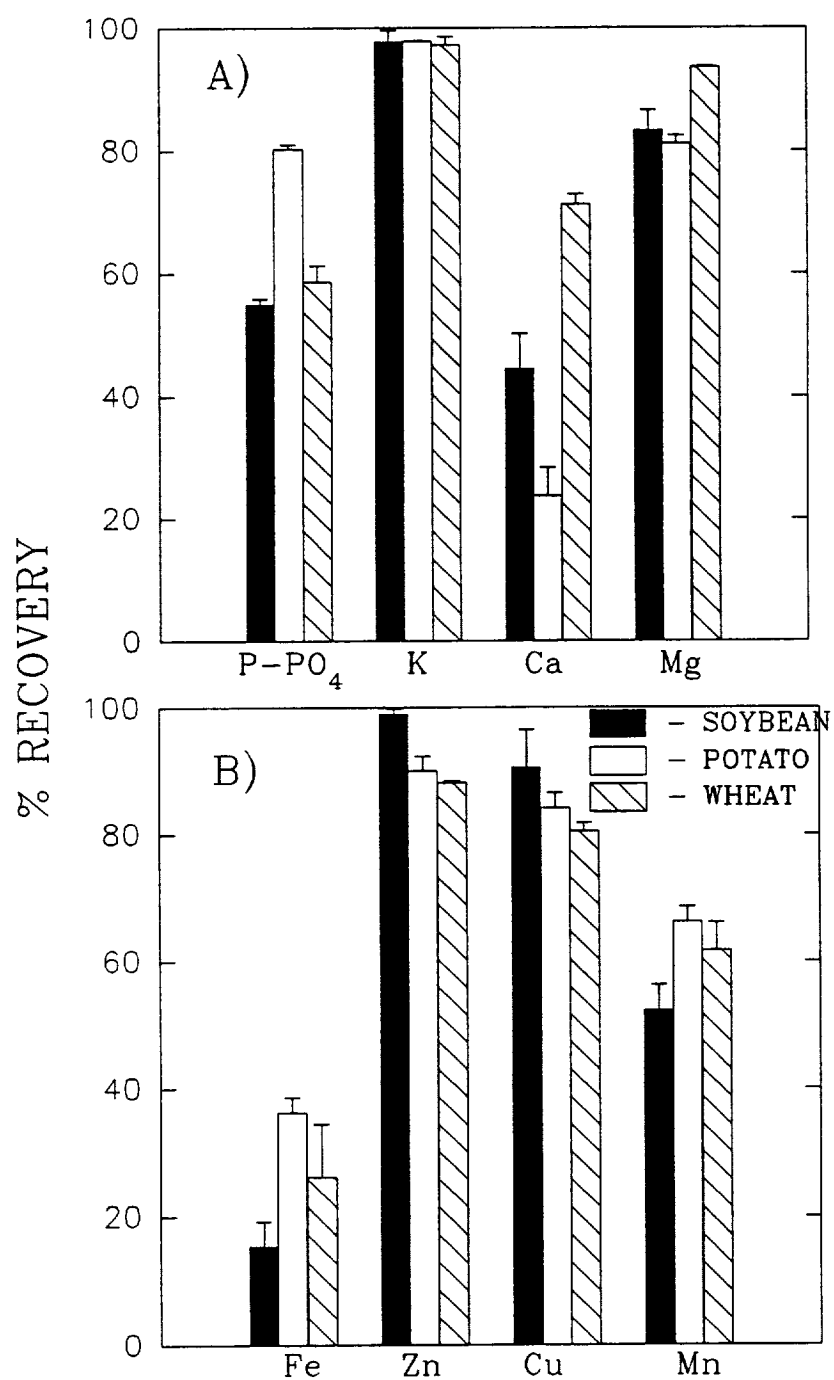


Figure 1. Percent recovery of nutrients from inedible plant biomass via leaching for A) macro-nutrients and B) micronutrients

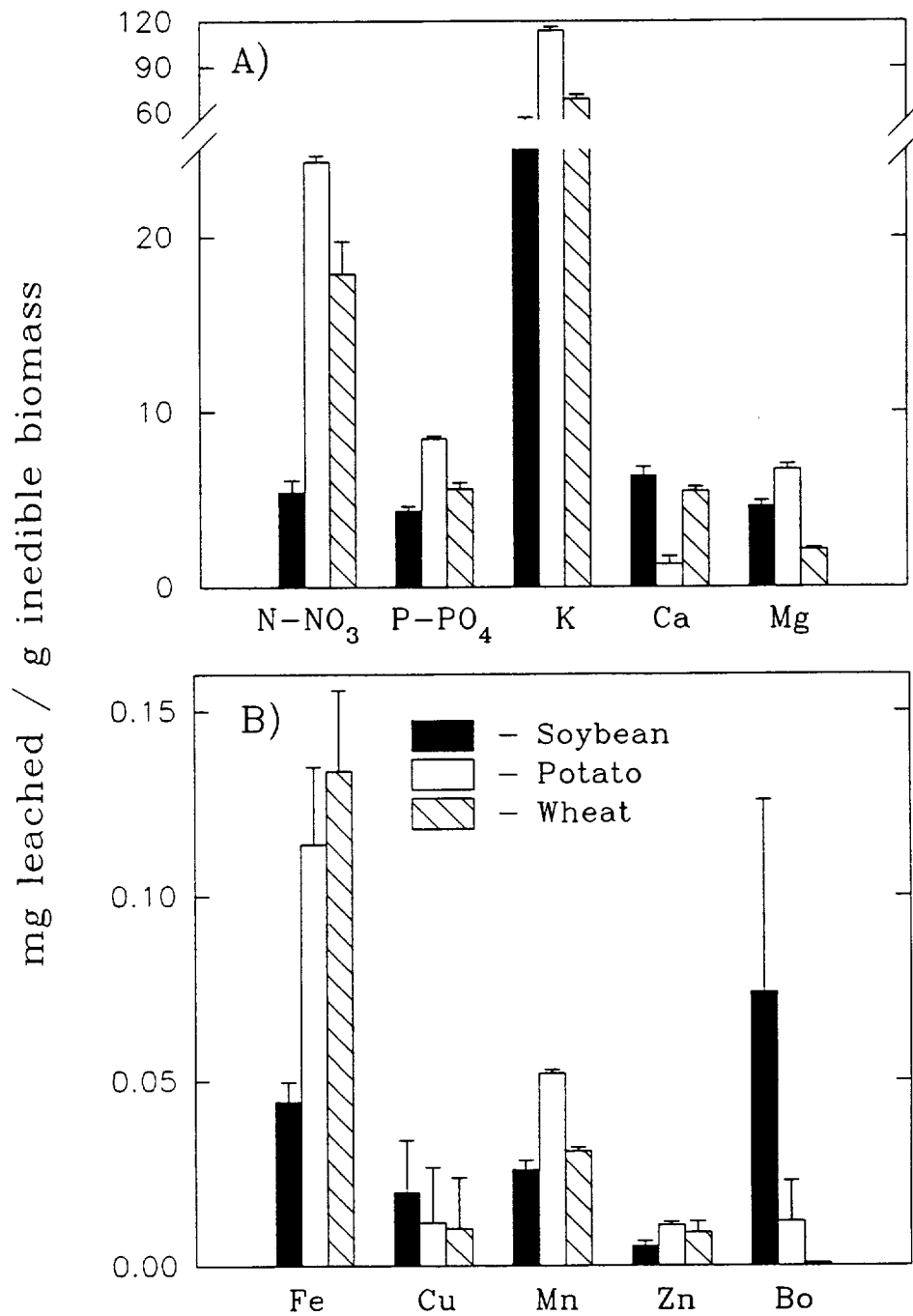


Figure 2. Amount of individual A) macronutrients and B) micronutrients extracted via leaching per unit weight of inedible biomass.

## Nutrient Requirements from Leachate

The relatively high recovery of nutrients by leaching suggests that the water soluble fraction of inedible plant material could be used as a significant source of recycled nutrients. An estimate of the total plant nutrient requirements that could be supplied by leachate was calculated as follows: 1) It was assumed that the vast majority of the nutrient requirements of wheat, soybean, and potato over the entire life cycle was supplied as part of replenishment solutions. 2) The total volume of replenishment solution added per plant growing tray to BPC-level growouts of wheat, soybean, and potato (11 L, 12 L, and 19 L, respectively) was known (C. Mackowiak, pers. comm.). 3) The nutrient content of the different crop leachates was compared to the standard KSC-CELSS replenishment solution (reference) (Figure 3). 4) The volume of leachate which could be produced per tray of BPC-grown wheat, soybean, and potato was estimated based on the inedible biomass produced per tray in the BPC (15 L, 10 L, and 5 L, respectively). 5) The ratio of leachate volume produced to replenishment solution volume required on a per tray basis was multiplied by the ratio of nutrient content in leachate relative to replenishment solution for each individual macro- and micronutrient.

Results indicate that recycling of the water soluble component of inedible crop residue can provide the majority of macronutrient requirements for wheat, but less than 50% for soybean and potato (Figure 4). The greater nutrient recycling potential via leaching for wheat is due to the larger percentage of inedible biomass (i.e. - lower harvest index), rather than greater leaching per unit biomass.

The ratio of nutrients within crop leachates is not the same as in a formulated hydroponic solution like 1/2 Hoagland's. Potassium levels tend to be higher relative to other macro nutrients, particularly for potato. In addition, micronutrient concentrations are lower relative to macronutrients. These nutrient imbalances suggest two approaches for formulating leachate-based nutrient solutions: 1) Diluting leachate to the point that no individual nutrient (i.e. - K) is in excess, and then supplementing with those nutrients that are at deficient level, or 2) using more concentrated leachate to minimize the level of supplementation, assuming that the concentration of some individual nutrients will be in excess of those found in 1/2 strength Hoagland's. As plant growth systems become fully integrated with resource recovery elements in future CELSS research, the concentration of individual nutrients in solution will be increasingly influenced by the chemical composition of different waste streams.

To aid in the development of leachate-based nutrient solutions, individual nutrient concentrations in all the different crop leachates are reported in Table 2.

## Ratio of Soluble and Insoluble Organic Material

A significant amount of the inedible biomass (29-52%) is readily soluble. By weight, the composition of this water soluble fraction is approximately 50% organic compounds and 50% inorganic nutrients (Figure 5). This estimate was produced by assuming that all soluble organic compounds were carbohydrates (i.e. - grams Carbon/L as determined by ultraviolet-assisted persulfate oxidation divided by 0.4 or



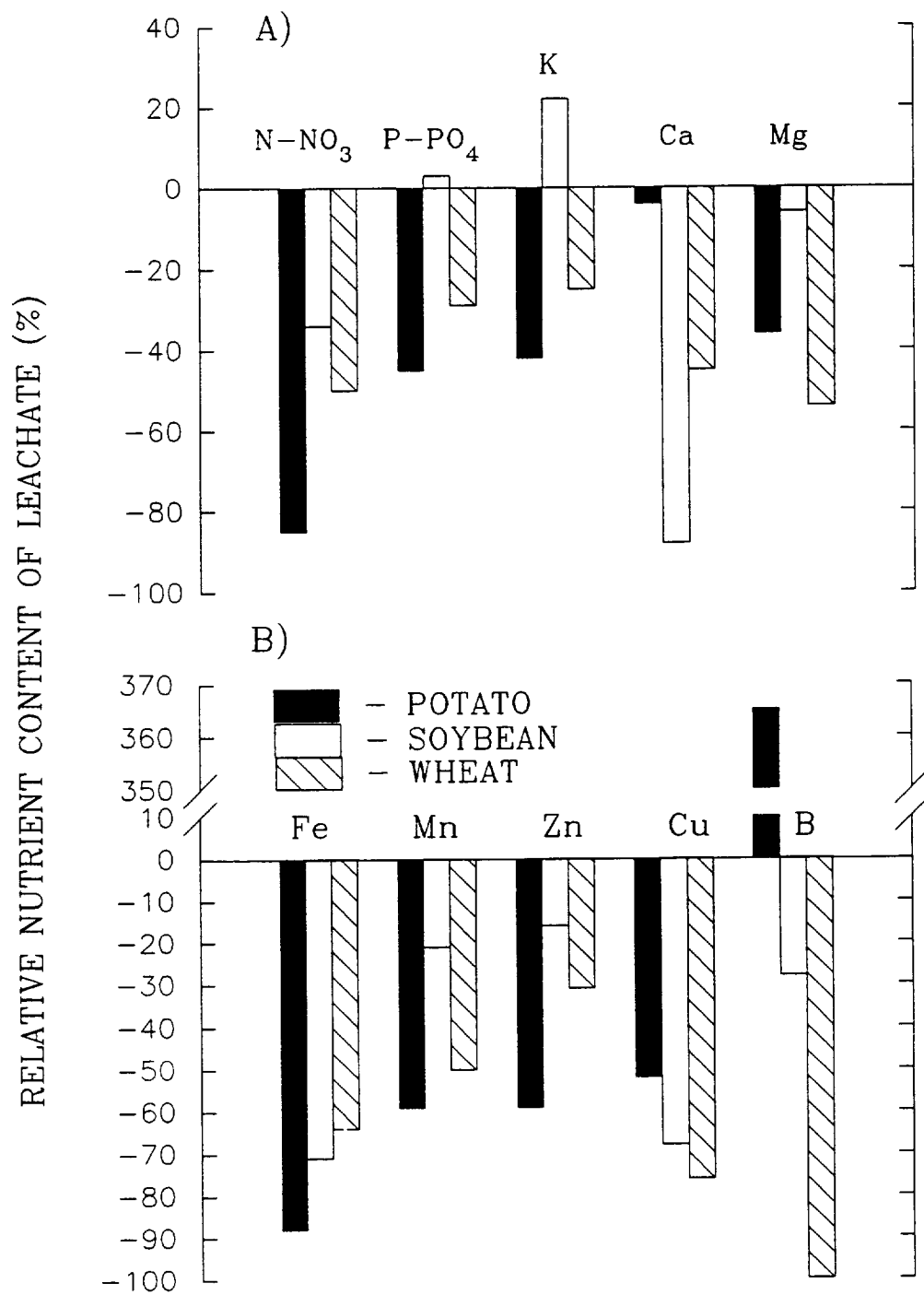


Figure 3. Nutrient concentration in water soluble fraction of inedible crop residue relative to concentration in standard replenishment solution.

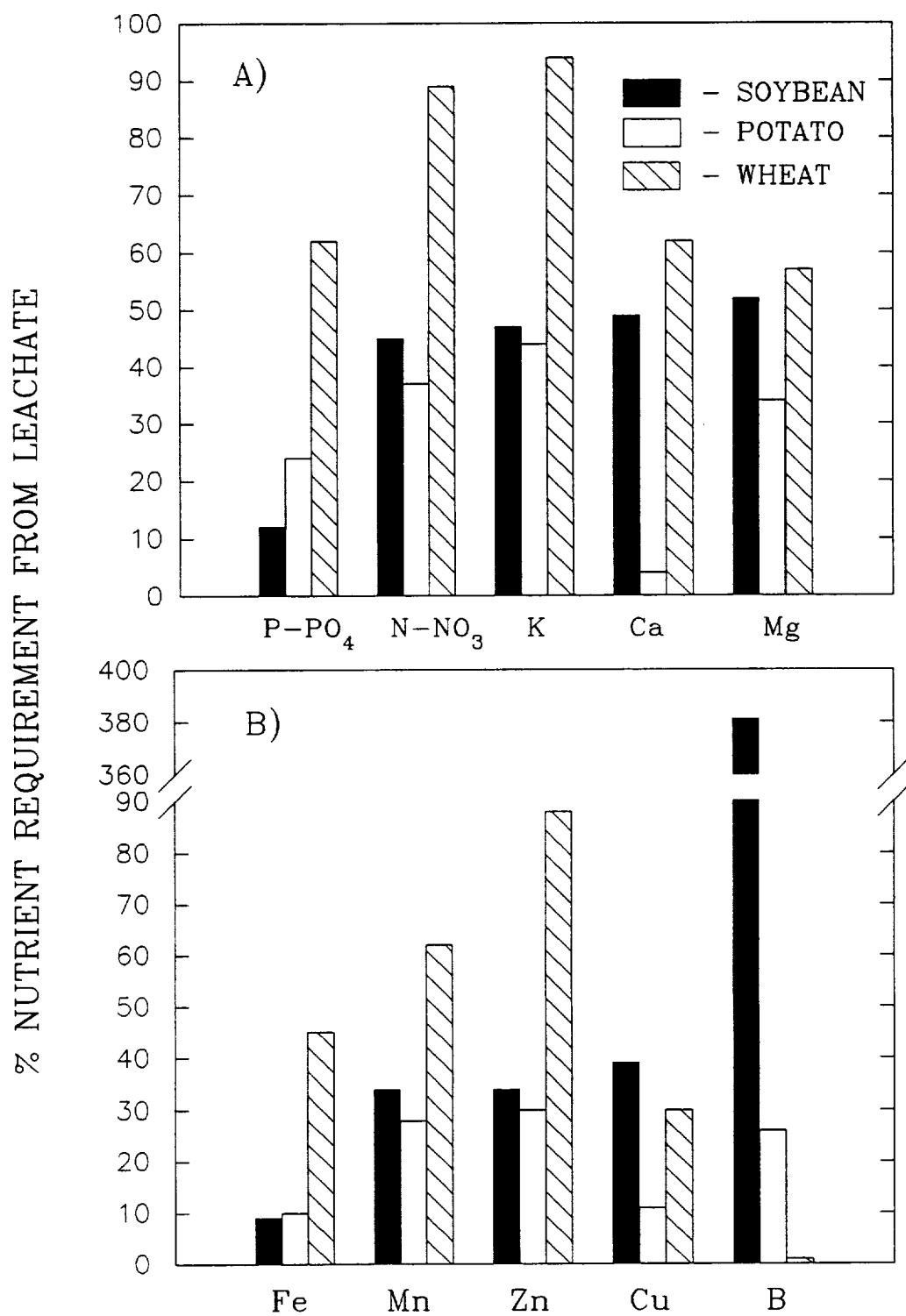


FIGURE 4. Percent of total nutrient requirements of different crops which could be supplied from the leachate of that crop. Values for A) macronutrients and B) micronutrients are based on both biomass production and nutrient utilization from Biomass Production Chamber studies.

Table 2. Chemical concentrations in different crop leachates.

Elemental Concentration	Crop Type		
	Wheat	Potato	Soybean
(mg/L)			
N-NO <sub>3</sub>	534(25.2) <sup>1</sup>	691(10.8)	160(19.5)
N-NH <sub>4</sub>	1.59(.08)	1.92(.07)	0.34(.11)
P-PO <sub>4</sub>	167(8.7)	240.6(4.4)	128(8.3)
K	2006(47)	3252(67)	1545(136)
Ca	163(5.6)	36.9(12.8)	187(14.7)
Mg	96.9(1.8)	190(9.0)	135(10.1)
Fe	3.98(.66)	3.26(.61)	1.31(.16)
(ug/L)			
Cu	303(408)	333(450)	595(413)
Mn	940(37)	1490(40)	765(74)
Bo	n.d.	344(337)	2180(1530)
Zn	271(91)	159(37)	323(23)

1- Values represents means and standard deviations of three replicate samples.

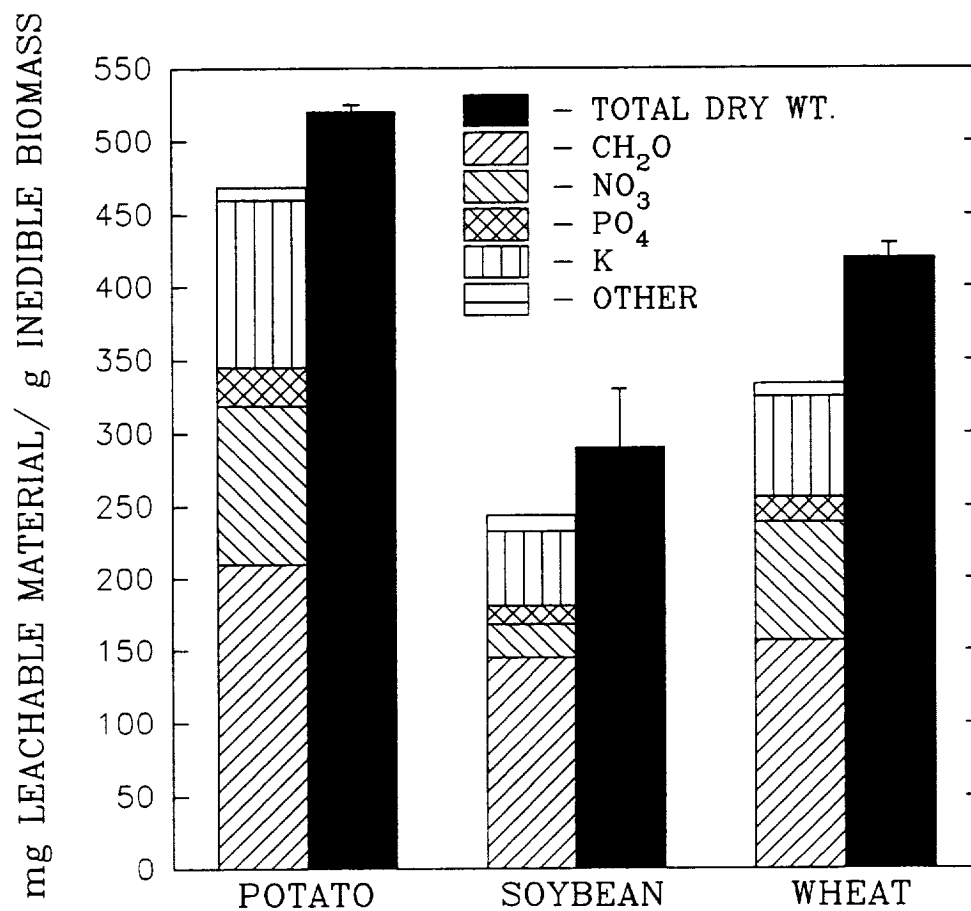


Figure 5. Composition of the water soluble fraction of inedible crop residue. Dry weight estimated from weight loss of biomass with leaching.

the percentage of the carbohydrate molecule comprised of carbon). Differences in the actual composition of the soluble organics may account for the discrepancy (8-9%) between the actual weight loss upon leaching and the sum total of weights of individual components measured in the leachate (Figure 3).

This estimated organic content of the leachate (50%) is lower than the 75-85% organic content (as estimated from ashing) of the original, unleached biomass, indicating that leaching can be viewed as a process which selectively removes inorganic nutrients from inedible biomass. Based on the assumptions that 1) all organic compounds are carbohydrate and 2) the leached biomass is composed solely of organic residue, approximately 25% of the organic content of inedible material is soluble.

One can view resource recovery in CELSS as a two-fold problem: 1) the recycling of nutrients required for plant growth, and 2) the conversion of carbon energy contained in undigestible forms into edible material. Leaching may serve as a simple, fast method of separating these two waste streams. This separation may be useful for a variety of reasons. If bioconversion of inedible material into edible products is deemed too costly or ineffective, leaching could be utilized to easily recover a large majority of nutrients in a form suitable for use in hydroponic systems prior to combustion of the organic-rich insoluble material. If a particular bioconversion process produces compounds which may be toxic to plants (e.g. - production of organic acids under anaerobic conditions), leaching could be used to separate a large percentage of the nutrients from the residue prior to processing.

Leaching can also be viewed as a process that separates the easily degraded, or labile, organic constituents of inedible residue from the more difficult to degrade, or recalcitrant, molecules. Preliminary experiments at KSC indicate that effective microbial digestion of the insoluble, polymeric plant material may require the presence of the labile, soluble fraction. Future research, therefore, may evaluate the liquid effluent, or supernatant, from biological reactors containing the entire inedible crop residue as a hydroponic solution.

### Summary and Conclusions

Leaching of inedible crop residues in water is an effective method to 1) recover the majority of inorganic nutrients contained in the biomass, and 2) separate the labile organic fraction from the more recalcitrant, insoluble organic material. In order to evaluate leaching as a component of the complete resource recovery scheme in CELSS, further studies are needed to evaluate 1) utilization of the inorganic nutrients by plants in hydroponic culture, and 2) conversion of soluble organics into microbial biomass for either direct consumption by humans or incorporation into the diet of intermediate, secondary consumers in the systems (e.g. - fish). When these data are obtained the viability of leaching can be judged in relation to other inedible crop material processing schemes.

## Acknowledgements

I would like to thank Wade Berry at UCLA, and Theresa Englert, Steve Black, and Steve Masokowski of the Bionetics Corporation at Kennedy Space Center for performing the chemical analyses on which this work is based. I am also grateful to Cheryl Mackowiak for providing plant production and nutrient requirement estimates from Biomass Production Chamber studies at the Kennedy Space Center. This work was performed under subcontract # 134-806-5 of NASA Contract # NAS10-10285.

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Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE Sept. 21, 1992		3. REPORT TYPE AND DATES COVERED NASA TM
4. TITLE AND SUBTITLE Characterization of the water soluble component of inedible residue from candidate CELSS crops			5. FUNDING NUMBERS	
6. AUTHOR(S) Jay Garland				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) The Bionetics Corporation Mail Code: BIO-2 Kennedy Space Center, Florida 32899			8. PERFORMING ORGANIZATION REPORT NUMBER 92-TM-02	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) NASA Mail Code: MD John F. Kennedy Space Center, FL 32899			10. SPONSORING / MONITORING AGENCY REPORT NUMBER TM 107557	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION / AVAILABILITY STATEMENT			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) <p>Recycling of inorganic nutrients required for plant growth will be a necessary component of a fully closed, bioregenerative life support system. This research characterized the recovery of plant nutrients from the inedible fraction of three crop types (wheat, potato, and soybean) by soaking, or leaching, in water. A considerable portion of the dry weight of the inedible biomass was readily soluble (29% for soybean, 43% for wheat, and 52% for potato). Greater weight loss from potato was a result of higher tissue concentrations of potassium, nitrate, and phosphate. Approximately 25% of the organic content of the biomass was water soluble, while the majority of most inorganic nutrients, except for calcium and iron, were recovered in the leachate. Direct use of the leachates in hydroponic media could provide between 40-90% of plant nutrient demands for wheat, and 20-50% of demand for soybean and potato. Further evaluation of leaching as a component of resource recovery scheme in a bioregenerative system requires study of 1) utilization of plant leachates in hydroponic plant culture, and 2) conversion of organic material (both soluble and insoluble) into edible, or other useful, products.</p>				
14. SUBJECT TERMS Nutrient recycling, bioregenerative, hydroponics, biomass processing			15. NUMBER OF PAGES 9	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT	



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